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MECHANICAL PROPERTIES OF SPRAY ATOMIZED SiC PARTICLE REINFORCED ALUMINUM-TITANIUM ALLOYS

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In the present study, the elevated temperature mechanical behavior and microstructure of Al-Ti-SiC_p metal matrix composites (MMCs) processed by spray atomization and co-deposition were investigated. The elevated temperature mechanical properties of the spray deposited and hot extruded materials were studied following a 100 hour exposure at 250, 350 and 450 °C; the room temperature mechanical properties were also determined. The results of the present study show that the elevated temperature yield strength of the spray deposited and extruded materials compared favorably to those of an equivalent alloy made by powder metallurgical materials, were superior to those of the ingot material, but were inferior to those of mechanically alloyed Al-Ti materials. The evolution of the microstructure as a result of the thermal annealing was investigated using X-ray diffractometry, TEM, and optical microscopy. The results from X-ray diffractometry studies showed extensive precipitation of the equilibrium Al_3Ti phase during thermal exposure. In addition, no interfacial reactions were observed in Al-Ti-SiC_p samples annealed at 500 °C for 24 hrs.

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FOREWORD

This report is the product of collaborative research performed at the University of California, Irvine (UCI) and the Naval Air Development Center (NADC). The work at UCI was supported by NADC Contract No. N62269-89-C-0242.

Introduction

Dispersion strengthened, elevated temperature aluminum alloys derive their strength and thermal stability from the presence of a dispersion of nanometer-size particles which effectively impede dislocation motion during deformation. The strengthening characteristics of these particulates at high temperatures are dependent on their ability to resist coarsening, and therefore low diffusivities and limited or no equilibrium solid solubility are desirable [1]. There are two processing approaches that can be utilized to synthesize aluminum alloys containing a dispersion of thermally stable, nanometer-size phases. The first approach involves energetically blending a mixture of fine aluminum alloy powders with a ceramic phase, typically oxides, carbides or nitrides, to produce a matrix containing a distribution of well dispersed, incoherent fine particles. Since these ceramic particles typically have no solubility in the aluminum matrix, they provide effective high temperature strengthening. The origin of this approach can be traced to work on *sintered aluminum powders* (SAP) during the late 1940's and early 1950's that eventually became the predecessor of various mechanically alloyed aluminum products investigated ever since [2, 3]. The second approach involves precipitating a fine dispersion of transition metal aluminide phases from the matrix through solid state reactions. Since the addition of transition elements to αAl under the near-equilibrium conditions present during ingot solidification commonly results in the formation of coarse, embrittling second phases as a result of their limited liquid and solid solubilities, these are usually added under rapid solidification (RS) conditions. The highly non-equilibrium conditions present during RS lead to the formation of extended solid solutions and metastable phases; the former can subsequently be decomposed into a fine dispersion of thermally stable precipitates [4]. The addition of transition elements to aluminum alloys, in combination with RS processing, has been successfully utilized to produce manifold aluminum alloys containing complex second phase dispersoids based on additions of Fe, Ce, V, Si, Cr, Mo, Ti and Zr [5-12].

Among the family of transition metal, high temperature alloys, Al-Ti alloys are actively being studied as a result of their attractive combination of elevated temperature properties [10-12]. These materials derive their excellent strength, ductility, and creep resistance from their fine grain structure and dispersion of Al_3Ti particles, in combination with the low solid solubility (0.8 at.%) and low diffusivity (3.86×10^{-15} at.% cm^2/sec.) of Ti in αAl [13]. In order to curtail the formation of coarse primary Al_3Ti, rapid solidification must be employed to extend the solid state solubility of titanium in aluminum. It has been found that 1.4 weight percent titanium (3.5 volume percent Al_3Ti) can be trapped in solid solution during gas atomization [14].

In an effort to further increase the modulus and strength of these materials, Al-Ti alloys are being studied as potential candidate matrix materials in metal matrix composites (MMCs) [15, 16]. The extreme reactivity akin to high additions of Ti, and the difficulties associated with the processing of MMCs have prompted the development of alternate synthesis approaches. One such approach, spray atomization and deposition, is actively being studied as a result of its ability to rapidly quench, reinforce and consolidate in a single step, thus avoiding the difficulties associated with the handling of fine, reactive particulates [15-18].

The objective of the present work was to provide preliminary results on the elevated temperature mechanical behavior of Al-Ti-SiC_p materials processed by spray atomization and co-deposition. In particular, the present work sought to establish the effects of the microstructure, as determined in a previous paper [16], on the resulting elevated temperature mechanical behavior.

Experimental

Two alloy compositions were used in the present study: Al-2.30 Ti and Al-4.03 Ti (in wt.%). The Al-2.30 Ti material was spray atomized and co-deposited with a distribution of 3 μm (d_{50}) SiC particulates (SiC_p), whereas the Al-4.03 Ti alloy was spray atomized and deposited in the absence of a reinforcing phase. An ingot metallurgy (IM) Al-6Ti material was also studied for

comparison purposes. A thorough discussion of the experimental details and processing variables can be found elsewhere [15-20]. For the reinforced material, the SiC_p co-injection distance was selected in the present study such that the atomized spray contained approximately 60% of the initial enthalpy; a discussion on the transfer of thermal energy during co-injection is available elsewhere [19].

Thermomechanical Processing and Mechanical Testing

Following spray atomization and co-deposition, the materials were subsequently hot extruded at 275 °C, using an area reduction ratio of 16:1. The hot extrusion step was used in the present study in order to eliminate the micron-sized porosity normally associated with spray atomized and deposited materials [20]. The Al-6Ti IM alloy was also hot extruded under identical conditions in order to provide a valid comparison with the spray deposited and hot extruded materials.

In order to investigate the effects of thermal exposure on the microstructure and properties of the as-spray deposited and hot extruded materials, the samples were encapsulated under an inert atmosphere and annealed at 250, 350 and 450 °C for 99 hours. The smooth bar tensile properties were determined according to ASTM E8-81, using 0.64 cm diameter specimens, in an Instron testing machine at a constant crosshead speed of 0.254 mm per second. The specimens were allowed to equilibrate at temperature for 1 hour, and subsequently tested under a tensile load; the room temperature properties were also studied. All of the annealing studies were carried out in an electrical resistance furnace under an inert atmosphere (Argon) with a temperature accuracy of $\pm$ 4 °C.

Microstructure

Optical microscopy was conducted on polished and etched, as-deposited samples using conventional and Differential Interference Contrast (DIC) techniques; the use of DIC microscopy facilitated identification of the SiC_p in the matrix. The samples were sectioned to a thickness of 0.5 cm, polished using conventional techniques, and etched with Keller's reagent.

Scanning Electron Microscopy (SEM) studies were conducted using a HITACHI S-500 microscope on as-spray atomized, and as-spray atomized and hot extruded samples. Image analysis (Dapple Prism system) of SEM micrographs was used in the present study in order to determine the effects of thermal exposure on the size and distribution of Al_3Ti precipitates in the matrix.

Transmission Electron Microscopy (TEM) studies were conducted using a JEOL 100C microscope at an operating voltage of 100kV, on samples of the unreinforced and reinforced materials. The TEM studies were carried out in order to investigate the effects of thermal exposure on the SiC_p/Al-Ti interface, and to provide insight into the coarsening behavior of the Al_3Ti particulates. The TEM samples were prepared using the window technique in a solution of 1:3::HNO_3:CH_3OH at 12 V and 1.5 ma; the solution was maintained at a temperature of -10 °C.

Phase Analyses

X-ray profiles of the Al-4Ti extruded material was generated on a Rigaku $\theta/2\theta$ powder diffractometer using a Cu tube operating at 45 KV and 15 ma. The lattice parameter of the Al-Ti solid solution was extrapolated from the Nelson-Riley (N-R) function, assuming that the absorption factors of the Al matrix and the Al_3Ti phases were identical [16]. The advantage of using the N-R function lies in its ability to incorporate both low and high incidence angles. Once the lattice parameter of Al-Ti solid solution was determined, the amount of Ti in solid solution was estimated from the data of Tonejc and Bonefacic [21]. Finally, the relative

intensity of the Al_3Ti peaks were obtained by dividing the height of the $(113)_\gamma$ peak by the height of the $(111)_\alpha$ Al peak.

Results and Discussion

Elevated Temperature Mechanical Behavior

A summary of the elevated temperature mechanical behavior of the spray atomized, deposited and hot extruded Al-Ti alloys (SD) is shown in Table 1 and Figure 1, and compared to those of equivalent materials prepared by powder metallurgy (PM) and mechanical alloying (MA). The results shown in this table for the Al-4Ti and Al-2.3Ti-SiC_p materials were obtained after a 99 hour anneal, followed by mechanical testing at the designated temperature. The results shown in Table 1 and Figure 1 show that the elevated temperature properties of the spray deposited materials compare favorably to those of the powder metallurgical materials, are superior to those of the ingot materials, but are inferior to those of the mechanically alloyed material. The excellent mechanical properties of the mechanically alloyed materials can be attributed to the development of a fine dislocation-precipitate (oxides and carbides) network during alloying, that effectively stabilizes the microstructure during deformation [5-8, 10]. The higher thermal stability of the spray atomized and deposited materials, relative to that noted for the cast ingot and extruded material, can be attributed to the faster quench rates, with concomitant microstructural refinement resulting from the spray deposition stage. This is supported by the sharp reduction in grain size and the absence of primary Al_3Ti phase observed for the as-spray deposited reinforced material, relative to that of the as-cast Al-Ti ingot.

Table 1: Elevated Temperature Mechanical Properties of Al-Ti-SiC Materials.

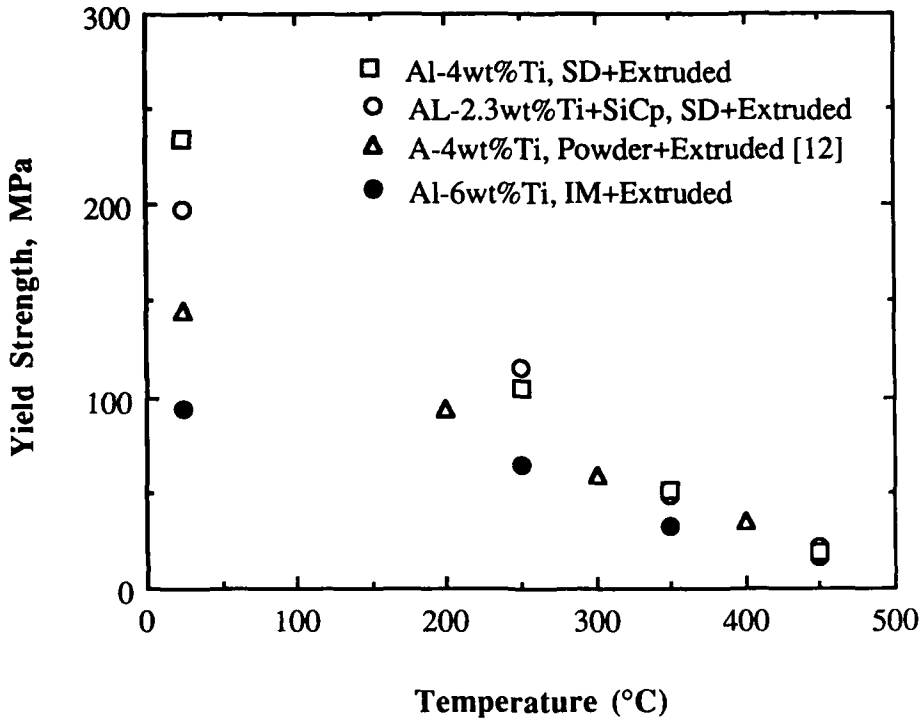
Material	Processing	Temperature °C	σ_{UTS} (MPa)	σ_{YS} (MPa)	Elong. (%)
Al-2.3Ti-SiC_p	SD[1]	25	200	198	7
		250	130	116	11
		350	51	48	18
		450	24	22	32
Al-4.0Ti	SD[2]	25	250	235	7
		250	127	106	24
		350	57	51	23
		450	20	18	40
Al-4.0Ti	PM[3]	25	180	145	23
		200	100	95	22
		300	65	59	30
		400	42	35	30
Al-4.0Ti	MA[4]	25	–	320	–
		160	–	280	–
		240	–	190	–
		290	–	170	–
		350	–	150	–

[1]Spray atomized, deposited and hot extruded materials. The SiC particulate size used here was 3 μm; the volume fraction was 6-8%.

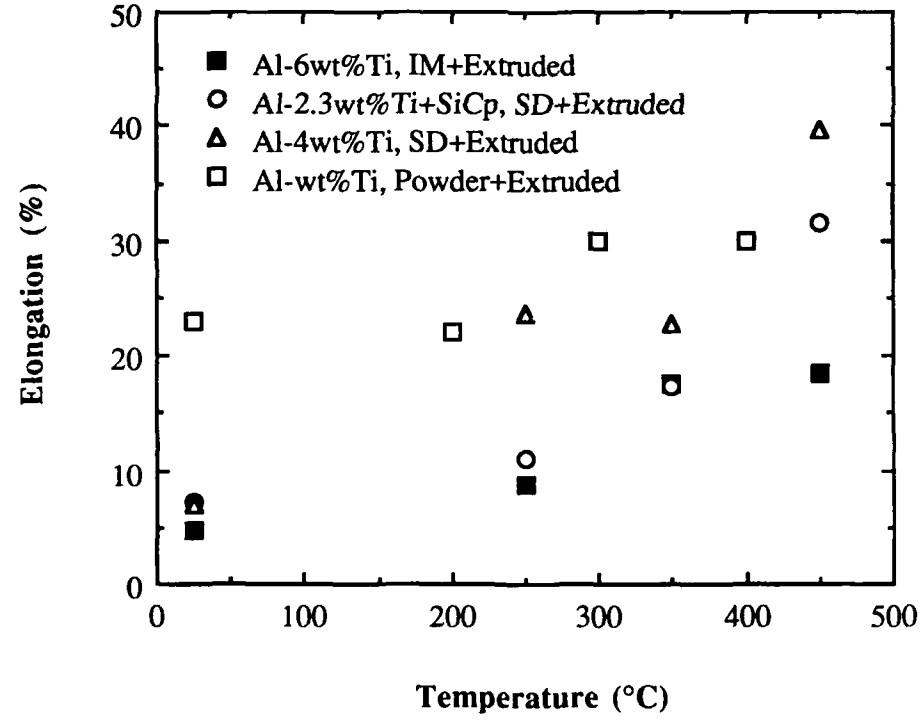
[2]Spray atomized, deposited and hot extruded unreinforced materials [12].

[3]Material prepared by powder metallurgy [12].

[4]Material prepared by mechanical alloying [12].



(a)



(b)

Figure 1. Comparison of the elevated temperature mechanical behavior of the spray atomized Al-Ti alloys to those of similar materials processed by powder metallurgy, and ingot metallurgy; (a) yield strength, and (b) elongation values after a 100 hour thermal exposure.

Inspection of the σ_Y values from Table 1 shows that at 250 °C the MA and SiC containing SD materials retained 60% of their room temperature yield strength, whereas the unreinforced SD and PM materials retained only 45-50%. The higher thermal stability of the SD material, relative to that obtained with the PM material, can be attributed to the coarser grain size of the former (20 μm) relative to that of the latter (1-5 μm). In addition, it is likely that the formation of a dislocation network in the vicinity of the $\alpha Al\backslash SiC_p$ interface will enhance the thermal stability of the SD material . Numerous studies have shown that although the dislocation density found in as-quenched, age-hardenable Al alloys is low, typically less than 10^5/cm [22], the dislocation density in reinforced Al matrices is on the order of 10^{13}/cm [23]. These dislocations, which are generated in order to accommodate the thermal mismatch strains associated with the differences in the thermal coefficient of expansion of the matrix and the reinforcement, are located primarily at the reinforcement-matrix interface, and decrease with increasing distance from the interface [23]. Finally, inspection of the σ_Y values obtained at 350 °C and shown in Table 1 suggests that, except for the mechanically alloyed material (0.46 $\sigma_{Y,\,RT}$), all of the alloys have lost a substantial fraction of their room temperature yield strength (0.20-0.30 $\sigma_{Y,\,RT}$) at this temperature.

The higher room temperature strength of the unreinforced SD, relative to that of the reinforced SD material can be attributed to the higher concentration of Ti of the former (4 wt.%) relative to that contained in the latter material (2.3 wt.%). The presence of a distribution of nanometer size Al_3Ti particles in the unreinforced material will provide more effective strengthening relative to that which can be obtained with micron size SiC_p in the reinforced material. This is evident from the Orowan Equation., where the flow stress for a metal strengthened by a dispersion of secondary phases can be estimated from:

$$\tau = \tau_m + \frac{Gb}{l} \qquad (1)$$

where τ_m in the matrix flow stress, G is the modulus of rigidity, b is the Burgers vector, and l in the planar center to center spacing between the dispersoids. Since interparticle spacing, l, is inversely proportional to particle size, a finer dispersoid will provide more effective strengthening.

It is worthwhile noting that the room temperature flow stress of the spray deposited material was higher than that obtained for the powder metallurgical material, at the expense of elongation. This is unexpected in view of the fact that, on the basis of quench rates, the grain size of the powder metallurgical material should be substantially smaller than the grain size resulting from spray atomization and deposition. This behavior, although not clearly understood, is thought to be related to crystallographic induced strengthening resulting from the small diameter extrusions.

In view of the relatively large amounts of elemental Ti present in both alloys, X-ray diffractometry studies were conducted in order to determine wether the Ti was retained in solid solution or present as a secondary phase; this is discussed further in the following section.

Microstructure and Phases

The as-spray atomized and deposited microstructure has been described elsewhere [15, 16] and was reported to consist of equiaxed grains in the 10-20 μm size range. Chanda et al. [16], used X-ray diffractometry to show that the spray atomized and deposited Al-4.0 wt.% Ti material retained 1.5 wt.% Ti in solid solution, whereas the as-spray atomized and deposited Al-2.3 wt.% Ti-SiC_p material retained up to 2.3 wt.% Ti in solid solution. Therefore, the presence of the Al_3Ti phase was readily discernable in the microstructure of the as-spray deposited

unreinforced material, as shown in Figure 2. In contrast, the presence of the Al_3Ti aluminide phase in the reinforced material became evident, only after extensive thermal annealing. This is consistent with the mechanical testing results which showed that the room temperature yield strength of the unreinforced material was higher than that noted for the reinforced alloy. The Al_3Ti particles present in the as-spray deposited unreinforced material were approximately 0.6 μm in size, and were randomly dispersed throughout the Al matrix. Figure 3 provides a high magnification view of an Al_3Ti particle surrounded by a well developed dislocation network in the hot extruded unreinforced material. The Al_3Ti aluminide phase can be formed by both primary solidification and peritectic transformation [25]. A peritectic phase transformation occurs at 665 °C and 1.15 wt.% Ti [34]: $L + \tau(Al_3Ti) \Rightarrow \alpha Al$. The precise wt.% Ti contained in the first solid to form is reported to be between 1.15-1.3 % [26, 27]. The coarse plate-like morphology shown in Figure 3 suggests that this particle evolved from a properitectic precipitation reaction in the temperature range of 1000 °C to 665 °C, and was attributed to the high amount of Ti present in the unreinforced material.

Following hot extrusion and thermal annealing (20 hours at 500 °C), the plate-like Al_3Ti precipitates coarsened substantially, and their principal axis became aligned parallel to the extrusion direction (see Figure 4). The plate-like phases are readily discernable in the SEM micrograph shown in Figure 4b. The extent of microstructural coarsening during annealing can be appreciated from Figure 5, where the average Al_3Ti particle size is plotted as a function of annealing time at 500 °C. In addition to coarsening, precipitation of the Al_3Ti phase from the supersaturated aluminum matrix during thermal exposure of the hot extruded reinforced material was evident. Since the lattice parameter of αAl changes only slightly with the addition of Ti, the results from the annealing studies are shown in Figure 6 as the relative intensity of the Al_3Ti peaks, obtained by dividing the height of the $(113)_\gamma$ peak by the height of the $(111)_\alpha$ Al peak [34]. The results shown in this figure are consistent with the previous discussion on thermal stability, since an increase in the relative intensity of the Al_3Ti peak corresponds to the observed decrease in properties with strength.

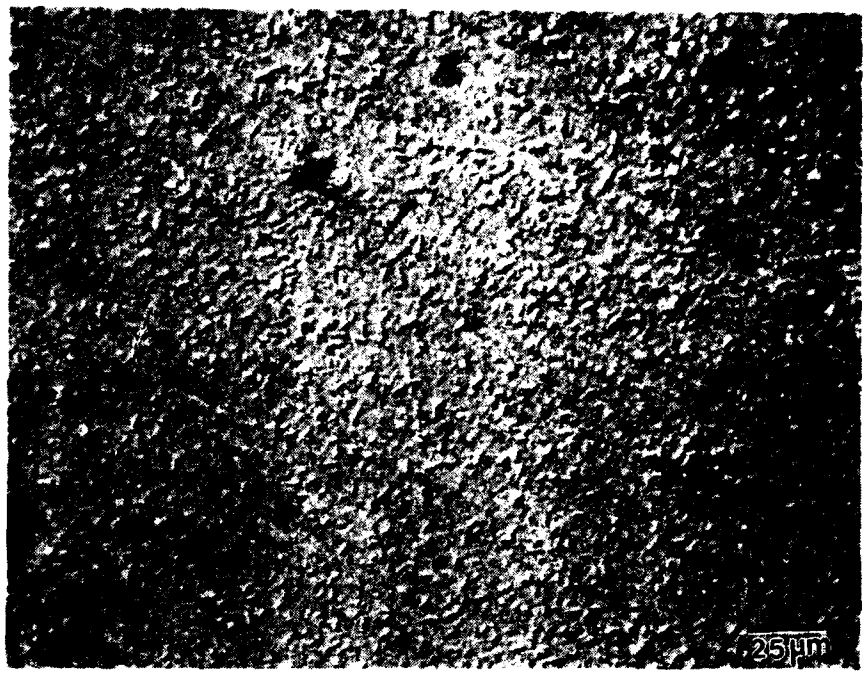


Figure 2. Optical micrograph showing the microstructure of the as-spray atomized and deposited unreinforced Al-4Ti material.

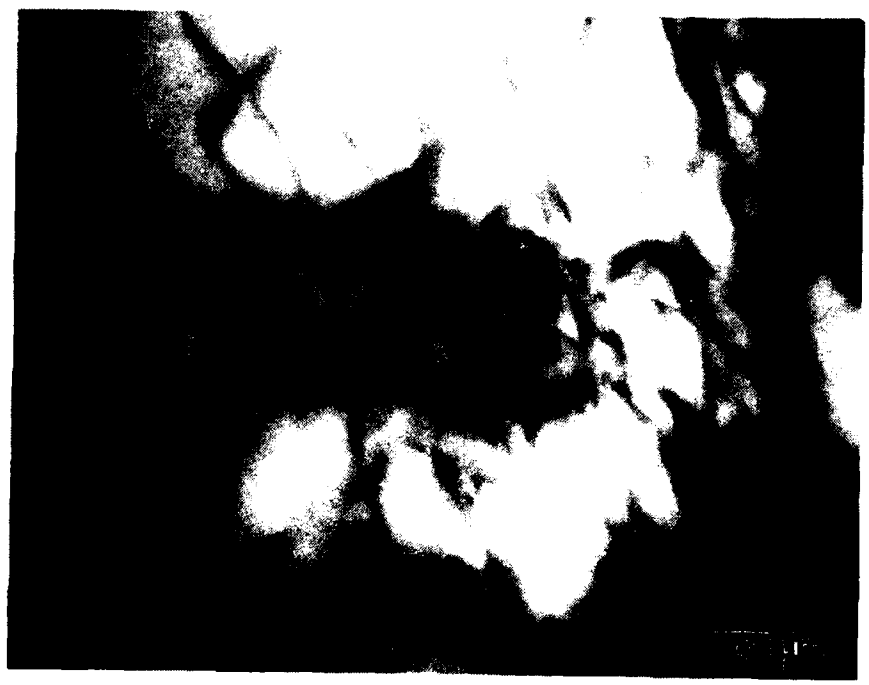
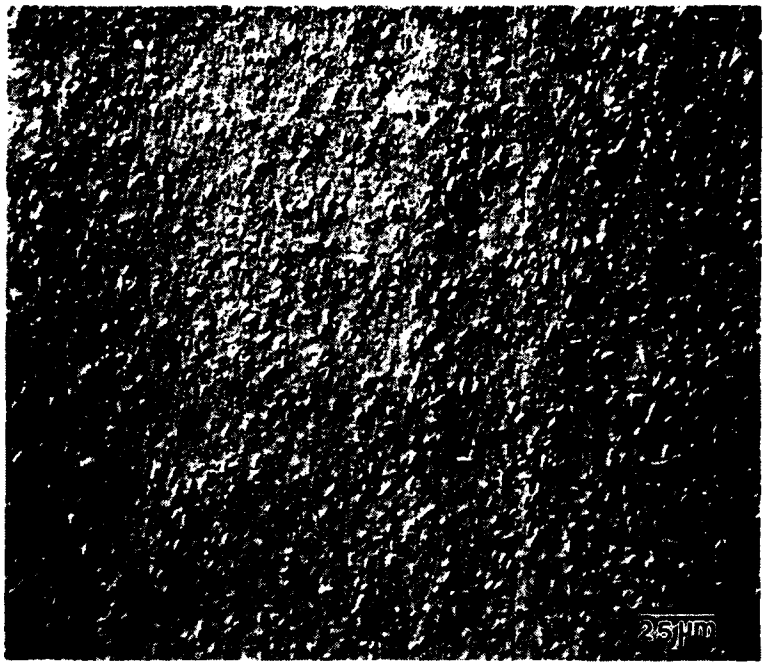


Figure 3. TEM micrograph of the as-spray deposited and hot extruded unreinforced material showing the precipitation of Al_3Ti particles.



(a)

(b)

Figure 4. Optical micrograph showing the microstructure of the as-spray atomized, deposited and hot extruded unreinforced Al-4Ti material, following a thermal exposure of 200 hours at 500 °C; (a) optical micrograph, and (b) SEM micrograph.

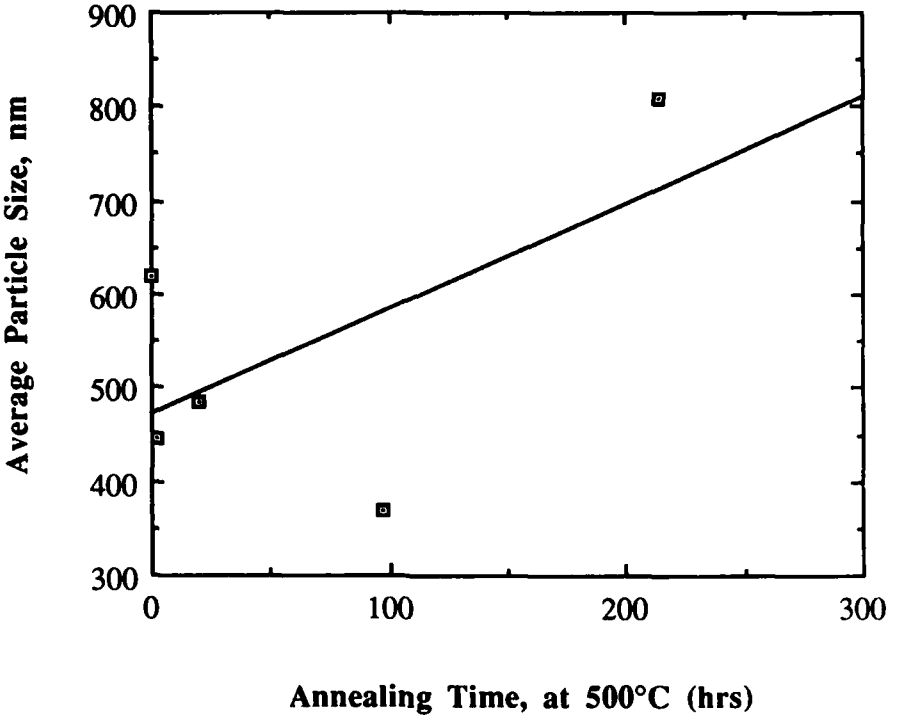


Figure 5. Variations in the average size of the Al_3Ti precipitates with thermal exposure at 500 °C in the unreinforced hot extruded material.

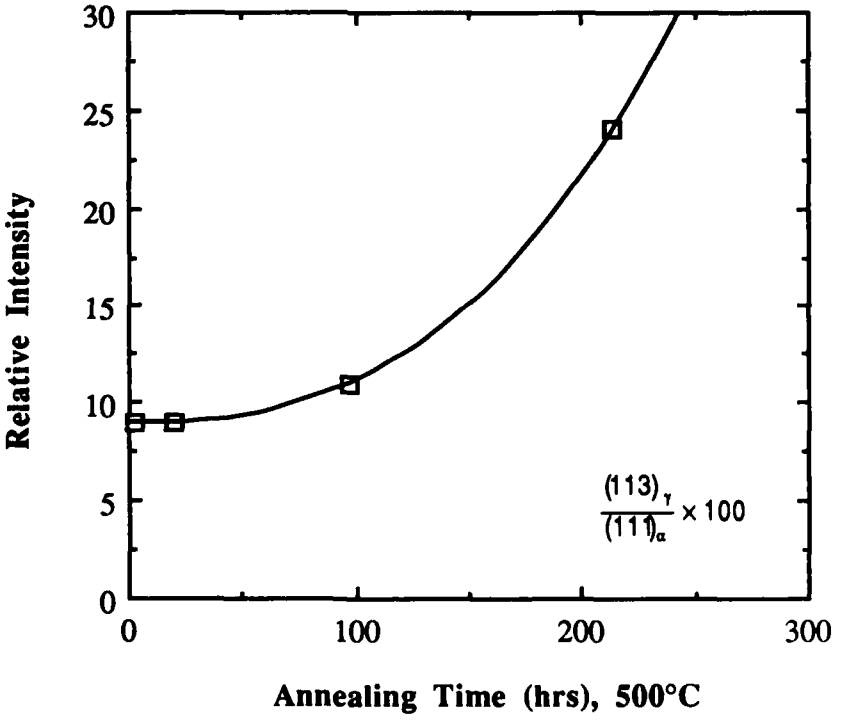


Figure 6. The effect of annealing temperature on the relative intensity of the Al_3Ti peak in the spray deposited and hot extruded unreinforced material .

Interfacial Behavior

In order to provide insight into the stability of the Al-Ti/SiC_p interface, the as-spray atomized reinforced material was exposed to an extreme thermal anneal of 24 hrs at 500 °C. TEM studies of the annealed samples failed to reveal the presence of reaction products at the interface. This is evident from the TEM micrographs shown in Figure 7, were the surface of the SiC_p remains relatively smooth, even after the extended thermal exposure, suggesting little or no interfacial activity. In the Al/SiC system, interfacial phases such as Al_4C_3 and Al_4SiC_4 normally form, either as a continuous layer or isolated precipitates [28]. SiC is known to react with molten Al ($T > T_m$) according to the reactions:

$$4Al + 3SiC = Al_4C_3 + 3Si \quad (2)$$

$$4Al + 4SiC = Al_4SiC_4 + 3Si \quad (3)$$

Further growth occurs, in the case of the first reaction, by solid state diffusion through the Al_4C_3 layer, and in the case of the second reaction, by the dissolution of the SiC into liquid Al. It is worthwhile noting that the latter reaction is anticipated on the basis of thermodynamics, however, the presence of the Al_4SiC_4 has yet to be confirmed experimentally. Although the bonding between SiC and Al_4C_3 is reportedly strong, the Al_4C_3-SiC interface is generally rough and can lead to regions of stress localization. For example, in graphite/Al system, degradation of the graphite reinforcement also occurs by the formation and growth of Al_4C_3 at a fast rate above 550 °C. Although no interfacial reactions at the Al-Ti SiC_p interface were evident after annealing, careful TEM studies did reveal extensive precipitation of the Al_3Ti phase. These are readily discerned in Figure 7b as 0.5-1 μm ellipsoids surrounded by a dislocation network.

Conclusions

The results of the present study show that the microstructure of an atomized and spray deposited Al-2.3Ti-SiC_p material consisted of an extended solid solution of Ti in αAl, whereas extensive precipitation of the equilibrium Al_3Ti phase was noted in an Al-4.0Ti unreinforced alloy. In addition, the present results show that the elevated temperature properties of the spray deposited and extruded materials compared favorably to those of an equivalent alloy made by powder metallurgical materials, were superior to those of the ingot material, but were inferior to those of mechanically alloyed Al-Ti materials. The difference in mechanical behavior was discussed in reference to the microstructural differences noted among all three types of materials. The results from X-ray diffractometry studies showed extensive precipitation of the Al_3Ti phase during thermal exposure. In addition, no interfacial reactions were observed in Al-Ti-SiC_p samples annealed at 500 °C for 24 hrs.

(a)

(b)

Figure 7. TEM micrographs of the as-spray deposited and annealed Al-Ti-SiC_p material (500 °C for 24 hours) showing: (a) the Al-Ti/SiC_p interface, and (b) precipitation of Al_3Ti particles.

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